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# United States Department of Agriculture, U. S. BUREAU OF CHEMISTRY,

H. W. WILEY, Chief of Bureau.

## *MODEL DENATURED ALCOHOL DISTILLERY*

### INTRODUCTION.

The model denatured alcohol distillery which is described in the following pages has been erected in accordance with the act of May 23, 1908, which authorizes the Secretary of Agriculture "to demonstrate and illustrate the methods for the making of denatured alcohol on a scale suitable for utilization by the farmer, or associations of farmers;" and it is operated under the provisions of Circular No. 721 of the Bureau of Internal Revenue, which exempt "distilleries having a daily spirit-producing capacity not exceeding 100 proof gallons, established by the Department of Agriculture at any of its experiment stations" from many of the "provisions of existing law relating to distilleries." A detailed description of the plant and its operation can not be given in the brief space afforded by this circular; still less, a guide to the practice of distilling. The circular will serve, however, to answer in advance many of the questions which are bound to arise in the minds of those who witness the operation of the plant.

### DESCRIPTION OF PLANT.

The distilling plant, which in its present form is specially adapted to the manufacture of denatured alcohol from maize or other grains, includes the following pieces of apparatus: A cylindrical iron cooker, a vacuum pump, four wooden fermenting tubs with the necessary connections, a beer pump, the distilling apparatus proper, and copper kettles and cans for use in making yeast.

*The Cooker.*—This piece of apparatus is a horizontally placed cylindrical vessel of about 130 gallons capacity, made of fairly heavy boiler iron, and is about  $2\frac{1}{2}$  feet in diameter and 4 feet in length. It is provided at the center of each end with stuffing boxes through which passes a shaft which carries a pulley outside the shell and stirring arms, known technically as "rakes," inside. In addition, provision is made in the ends for inserting thermometers and for drawing samples of the contents for testing.

In the center of the top of the cooker is a dome closed with a man-hole plate and entered by a pipe connection leading to a "cross." From one side of the cross connection is made to the steam supply, which can be carried as high as 55 pounds pressure; from the other side connection is made to the vacuum pump; and from the top a pipe is led to a blow-off valve. At one end of the cooker, and on top, is set

a metal funnel of about 10 gallons capacity, which opens into the shell through a suitable valve.

From the under side of the cooker a draw-off pipe leads to the fermenting tubs, the whole apparatus being raised on a platform sufficiently above these tubs to secure complete drainage into them.

The cooker is used for boiling the raw grain, under pressure, until its starch has been gelatinized into a thick paste, suitable for further treatment with malt.

*Vacuum Pump.*—This apparatus is of the “wet vacuum” type, with jet condenser, and is used to reduce the pressure within the cooker below that of the atmosphere, and thereby to lower the temperature of the contents of the cooker. An abundant supply of cold water is necessary to its operation.

*Fermenting Tubs.*—There are four of these tubs, each being 36 inches in diameter and 30 inches deep and having a total capacity of 132 gallons. They are filled in working only to a depth of 27 inches, corresponding to a capacity of 120 gallons. The space of 3 “dry inches” is allowed for the froth and foam which are thrown to the surface of the fermenting liquor. A fair grade of pine would be a suitable material for these tubs in a denatured alcohol distillery.

These “fermenters” are raised about a foot off the floor, on strong joists, and are provided underneath with outlet valves and copper discharge pipes which lead to the beer pump. Under the regulations of the Bureau of Internal Revenue these pipes must be painted red, being intended for the conveyance of beer. They must pitch toward the beer pump, and must be free of pockets, in order that they may be washed and drained thoroughly.

*Beer Pump.*—For a temporary installation, like the present one, a common iron steam pump serves very well. A permanent installation would call, on the contrary, for a brass-end pump which would be better suited to resist the corrosive action of the fermented mash. The valve action of the pump should permit considerable variation in its speed, as otherwise trouble might arise in feeding mash to the still at just the proper rate.

*Distilling Apparatus.*<sup>1</sup>—This, the most complicated and most costly part of the distillery plant, consists of—

<sup>1</sup> The cut on the opposite page represents the interior construction of a still built on the same principles as the one described above, although larger and provided with more chambers. The parts of the two columns are numbered as follows:

	Beer still.	Alcohol column.
Kettles, with steam inlet (or coil) .....	1	2
Chambers, with trapped overflows .....	3	4
Perforated copper floors .....	5	--
Solid floors with boiling caps .....	--	6
Beer-heating still head .....	7	--
Water-jacketed still head .....	--	8
Inlets for beer and alcohol vapor .....	9	10
Outlets for alcohol vapor .....	11	12
Condenser and try-box .....	--	13-14

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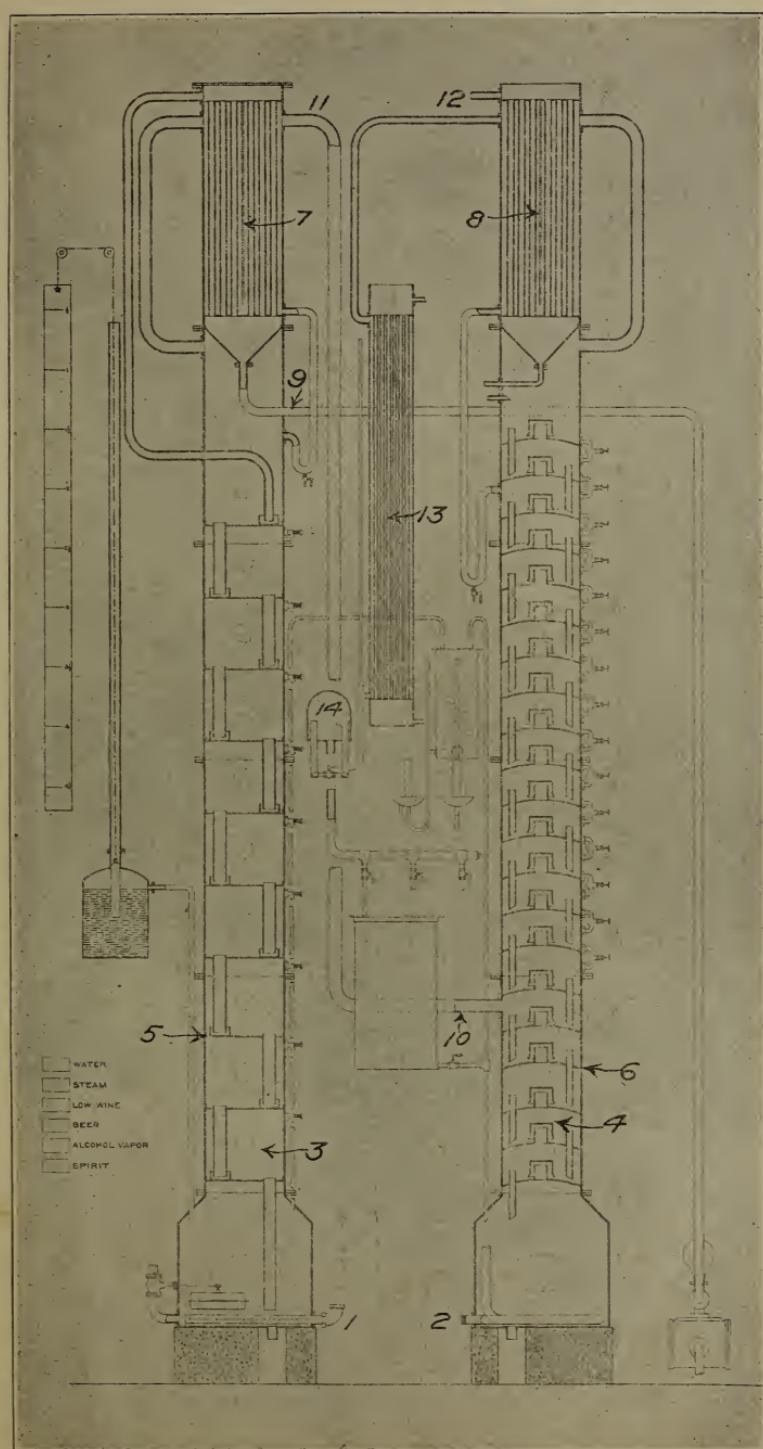


FIG. 1.—Special column still for manufacture of denatured alcohol.

1. A beer still in which the alcohol formed in the course of fermentation is liberated from the mash.
2. An alcohol column in which the vapors from the beer still are freed of most of the water which they contain on leaving that apparatus.
3. A condenser in which these same alcoholic vapors, substantially freed of moisture, are condensed and liquefied by contact with cold metallic surfaces.
4. Two alcohol tanks, one for the reception of strong spirit and the other for weaker spirit, known technically as "low wines."
5. A denaturing tank in which the finished alcohol of a strength not lower than 180° proof is mixed with authorized denaturants.

*Beer Still.*—In outward appearance this is a column about 6 feet high and 10 inches in diameter, standing on a base about 2 feet high and 20 inches in diameter, the whole apparatus being made of fairly heavy copper. The enlarged base, which is a sort of boiling kettle, is provided with a perforated steam inlet and a draw-off valve. The column proper is divided into two main parts by a horizontal diaphragm located about 2 feet from the top. The space above this diaphragm serves as a beer heater, and in effect is a tank, provided with beer inlet and overflow, and is penetrated by the outlet pipe through which the hot alcoholic vapors escape from the still. The space below this diaphragm, down as far as the enlarged base, is divided by horizontal partitions of perforated copper into eight small circular chambers, situated one above the other. The uppermost chamber is fed with fresh hot beer by the overflow pipe leading from the beer heater. Each chamber is provided with its own overflow, trapped at the bottom, through which its liquid contents escape into the chamber next beneath as soon as they reach a certain depth upon the floor of the chamber. The perforations in the floors between the chambers provide for the upward passage and escape of the steam and alcohol vapors liberated in the still, and at the same time are so small as to prevent beer running down through them when the apparatus is in operation. The beer inlet is painted red; the vapor outlet, black; the steam inlet, aluminum bronze.

*Alcohol Column.*—This structure closely resembles the beer still in dimensions and in general outward appearance, but is quite different as to certain details of internal structure. Like the beer still, it consists of a kettle at the base, a series of circular chambers, and a tank at the top, which is penetrated by the outflow pipe for strong alcohol vapor. Heat is supplied in the kettle by means of a closed coil, instead of by a perforated pipe. The individual chambers are provided with overflows, as in the beer still; but the floors between them are not perforated. Instead, they have large central openings, the edges of which are turned upward into short wide tubes which are covered and trapped

by inverted copper bowls known technically as boiling caps. The tank at the top of the column is supplied with an inlet pipe for cold water and with an overflow. These water pipes are painted white.

The large black pipe which enters the kettle brings weak alcohol vapors from the beer still to the alcohol column. The black pipe which passes from the top of the column to the smaller, centrally located condenser is the outflow for strong alcohol vapor.

*Condenser.*—This piece of apparatus consists essentially of a copper worm, placed within a tall, narrow copper tank. The tank is filled with cold water and the worm is submerged in it. The strong alcohol vapors, entering at the top of the worm, are condensed therein to liquid alcohol, which escapes at the bottom through the "try-box," which is provided with instruments for registering the strength of the spirit produced.

*Alcohol Tanks.*—The flow of spirit from the try-box may be diverted at will into either of these tanks. The one nearest the beer still is used for the collection of spirit of a strength not less than 180° proof (= 90 per cent alcohol, by volume, at 60° F.). The other, known as the low-wine tank, is used for the storage of spirit of lower strength, which must eventually be redistilled to raise its proof.

These two tanks, as well as the denaturing tank, are so constructed that all openings into them may be secured by Government locks. Removal of alcohol contrary to law, and fraud upon the public revenue are thus prevented.

#### OPERATION OF THE PLANT.

*Materials.*—The following materials are employed in this plant for demonstrating the manufacture of alcohol: Raw grain, including both maize and rye, finely ground; barley malt, crushed moderately fine; ice; and yeast, prepared fresh every day.

The method of manufacture is as follows:

*Mashing.*—The cooker is filled to about one-half its depth with water at a temperature just above blood heat, about 100° F. The stirring rakes are started and, the manhole plate being removed, 140 pounds of corn meal are run into the water so slowly and carefully as to prevent the formation of lumps containing dry meal in the center. Simultaneously the temperature is raised slowly to 130° F. All the corn having been added, the manhole plate is screwed down, and the full steam pressure (55 pounds) is turned on. In about half an hour, during which the rakes are run continuously, the temperature should have risen to about 300° F., at which point it is held for five minutes. Steam is now shut off, and the blow-off valve is opened. When the pressure in the cooker is relieved, the blow-off is closed, and the vacuum pump is started and run vigorously until the temperature within the cooker has fallen to 140° F.

While the cooking of the raw grain has been going on, fifteen pounds of good barley malt, crushed fine, have been macerated in enough warm water, at a temperature of  $120^{\circ}$  F., to form a fairly fluid mixture. The volume of this mixture should be between 8 and 10 gallons. This is added to the contents of the cooker at  $140^{\circ}$  F., and the mixture stirred vigorously at that temperature for about two hours. The action of the malt during this time converts the gelatinized starch of the grain into fermentable sugars. The mixture is next blown out into a fermenting tub, and is cooled to about  $65^{\circ}$  F. by adding ice.

*Yeast.*—Two days before beginning the operations described under the head of mashing the preparation of yeast is begun. Ten pounds of finely ground rye flour are mixed with sufficient water at a temperature of  $128^{\circ}$  F. to form a fairly thin paste, and then 10 pounds of fine crushed barley malt are added with stirring. The temperature of this mixture is then raised to about  $145^{\circ}$  F. and is held at that point for between one and two hours. The temperature is then lowered to about  $128^{\circ}$  F. and the mixture is allowed to stand for approximately twenty-four hours at that temperature, during which time it becomes quite sour from the formation of lactic acid, a substance which tends to protect the yeast from invasion by undesirable germs. The temperature of the mixture is now reduced to about  $70^{\circ}$  F. and a small quantity of selected seed yeast is added. At this time the gravity of the sour yeast mash should register about  $24^{\circ}$  on the Balling hydrometer. The yeast mash is now allowed to ferment until its gravity has fallen to between  $5^{\circ}$  and  $6^{\circ}$  Balling, which takes ordinarily somewhat less than one day. The yeast is now ripe and ready for addition to the main mash, prepared as described above.

*Fermentation.*—The gravity of the main mash after the addition of the yeast mash should register about  $10^{\circ}$  on the Balling hydrometer, and its temperature at the time of setting should be between  $63^{\circ}$  and  $70^{\circ}$  F. according to the temperature of the room in which the fermenting apparatus is situated. Fermentation, as indicated by the appearance of gas bubbles, begins in a few hours and is accompanied by a decided rise in temperature. Care should be taken, however, to keep the temperature from going above the limit of  $92^{\circ}$  F., as otherwise there is danger of the formation of acetic acid and of a loss of alcohol. According to the nature of the material employed, the vigor of the yeast, and the temperature at which the fermentation is conducted, the tub will be ready for distillation at the end of from forty-eight to sixty hours. Its gravity at this time should have fallen to about  $-0.8^{\circ}$  Balling.

*Distillation.*—The outlet valve of the fermenting tub containing the ripe beer is now opened and the beer is pumped slowly into the beer still, which previously has been charged with water and heated by blowing live steam into the kettle at its base. At the same time steam

is turned on through the coil in the kettle of the alcohol column and cold water is turned into the tank at the head of the alcohol column and into the condenser tank. Alcoholic distillate will presently appear at the overflow in the try-box and, according to the proof which it shows on the testing instruments in the try-box, it is turned either into the alcoholic tank or into the low-wine tank. It is the aim of the distiller, by regulating the supply of steam, of beer, and of cold water in relation to one another, to produce a spirit from the outset which will have a proof not lower than  $180^{\circ}$ . Failing to do this, he is obliged to collect his spirit of lower proof in the low-wine tank, after which it is returned through the alcohol column and redistilled until it has reached the proof of  $180^{\circ}$ , below which it can not be denatured legally.

*Denaturing.*—The contents of the alcohol tank having a proof not lower than  $180^{\circ}$  are run at the close of the day's distillation into the denaturing tank, the volume of the spirit is measured therein by the scale attached to the gauge glass, and authorized denaturants are added according to the formula prescribed by the Bureau of Internal Revenue. In the case of this model distillery these denaturants are approved wood alcohol and approved benzin.

From every bushel of grain (56 pounds) about  $2\frac{1}{2}$  gallons of strong alcohol should be secured.

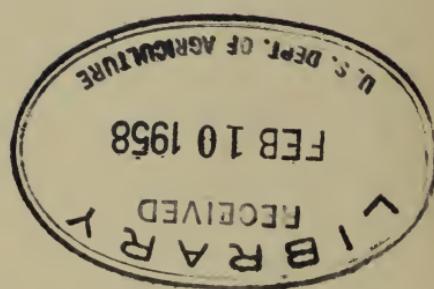
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WASHINGTON, D. C., December 1, 1908.







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